SOME USEFUL APPLICATIONS OF ZIRCONIUM

BY J. H. McCLAIN AND R. W. NELSON

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SOME USEFUL APPLICATIONS OF ZIRCONIUM

by

J. H. McClain[1/] and R. W. Nelson[2/]

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CONCLUSIONS

In the last 3 years' operation of the zirconium-production plant, zirconium metal has been employed to good advantage in many applications. The most frequent use has been in situations requiring resistance to attack by hydrochloric acid. In this service, it has been shown to be a serious competitor to Hastelloy.

Zirconium also has excellent resistance to most other corrosives and could be considered for use as an all purpose metal. For example, this report cites 18 applications in hydrochloric acid, 4 applications in sulfuric acid, 5 applications in phthalic-hydrochloric acid, 3 applications in wet hydrogen chloride-chlorine atmospheres, and 2 applications in fused caustic.

SUMMARY

Throughout the various processing steps necessary to the production of zirconium, there are many corrosive atmospheres, solutions, and conditions that defy the usual or normal materials of construction and make the Kroll process a useful proving ground for practical applications of zirconium.

In the past, ordinary steel construction was employed to expedite the fabrication of equipment needed to meet immediate production demands. In other instances, the finest material available was employed to meet definite conditions, with the knowledge that failure sometimes was inevitable over a period of time, in spite of careful selection.

Zirconium metal has been used with a high degree of success in solving these problems. This report describes those applications but does not attempt to evaluate the corrosion rates in the usual finite terms of the corrosion expert.

INTRODUCTION

In February 1947 the Bureau of Mines began to produce zirconium at the Northwest Electrodevelopment Laboratory, Albany, Oreg. The first pilot plant produced about 60 pounds of Kroll-process zirconium metal per week. In June 1949 a larger plant, having a nominal capacity of 300 pounds per week, was put into production. The present plant, which began operations in September 1950 and was expanded further in 1952, produces over 5,000 pounds a week.[3] This growth in production was the outcome of demand for the metal by the Navy Department and the Atomic Energy Commission.

Their use for zirconium metal has never been clearly stated, but its nuclear, chemical, and mechanical properties make it an ideal construction material for atomic power plants of the thermal type.[4][5]

3/ Shelton, S. M., and Dilling, E. D., Manufacture of Zirconium Sponge: chap. in Zirconium and Zirconium Alloys: Am. Soc. Metals, 1953, p. 82.

4/ Miller, E. C., Zirconium, A Structural Material for Nuclear Reactors: Nucleonics, vol. 11, No. 7, 1953, p. 27.

5/ Zinn, W H., Nucleonics, vol. 10, No. 9, 1952, pp. 8-14.

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During this period of production growth, very little zirconium was available for other practical uses at justifiable costs. Considerable work was done, however, in the corrosion laboratories, where it has been established that zirconium resists the action of all cold mineral acids, as well as alkalies at all temperatures and concentrations.6/7/8/

As zirconium became more plentiful, it was possible to obtain allocations of this metal for fabrication into plant replacement parts. Photographs of new parts and equipment were taken before installation, and case histories were begun. In some instances, it was possible to take pictures after some time had elapsed; in others, this was not feasible without interrupting production.

This report is a compilation of those applications and is broken down into three main classifications:

A. Experimental applications.
B. Production applications.
C. Laboratory and other.

EXPERIMENTAL APPLICATIONS

First Experimental Pulse Column

An experimental pulse column, designed by L. A Yerkes9/ and G. L. Frederic,9/ was operated for 6 months. This column was 21 feet high and employed zirconium plates 1/16 inch thick and 1-1/2 inches in diameter. Each plate had 84 - 3/32-inch drilled openings. A zirconium rod supported the plates at 2-inch intervals. The solutions used alternately and jointly in a system were as follows:

1 to 3 normal hydrochloric acid containing trace amounts of ferric chloride and 2 normal thiocyanic acid.

5 normal sulfuric acid.

A ketone solvent was present in all solutions and prohibited the use of most plastics for this application.

No visible evidence of corrosion was noted at the time the plant was dismantled.

Second Experimental Pulse Column

The zirconium parts of pulse column 1 were salvaged and incorporated in a second pulse column 16 feet high (fig. 1). This column has been used for various explorations, but the only one considered was its use in separating tantalum and columbium. The solution employed in this system was 12 normal hydrochloric acid and approximately 2.25 normal tantalum, columbium, and ferric chlorides. This test lasted approximately 1 month.

6/ Golden, Lex B., Lane, Roy I. Jr., and Ackerman, Walter L., Corrosion Resistance of Titanium, Zirconium, and Stainless Steel: Ind. Eng. Chem., vol. 44, No. 8, 1952, p. 1930.

7/ Materials and Methods, Comparison of Corrosion Properties of Zirconium, Titanium, Tantalum, Stellite No. 6, and Type 316 Stainless Steel: 1952, No. 221.

8/ Golden, Lex B., Corrosion Resistance of Zirconium; chap. in Zirconium and Zirconium Alloys: Am. Soc. Metals, 1953, p. 305.

9/ Chemical engineer, Northwest Electrodevelopment Laboratory, Bureau of Mines, Albany, Oreg.

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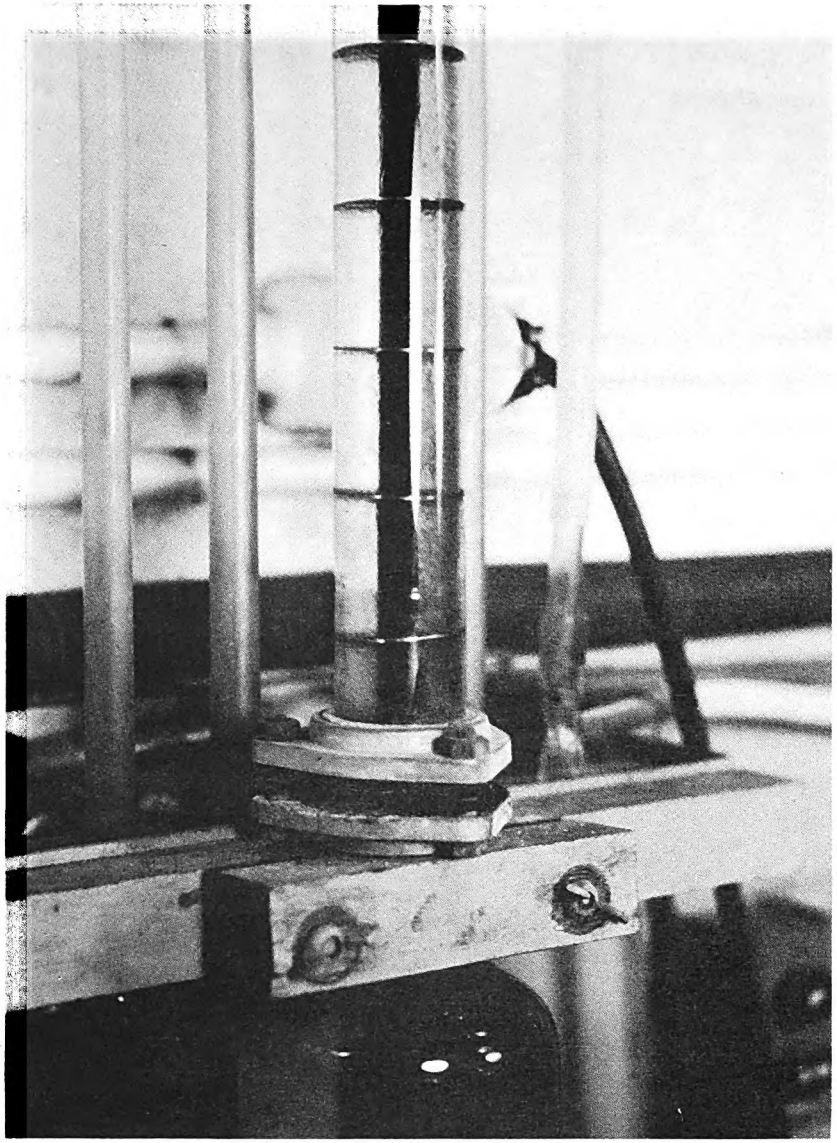

Figure 1. - Experimental pulse column, showing zirconium dispersion plates.

Figure 2. - Zirconium bomb designed for special corrosion studies.

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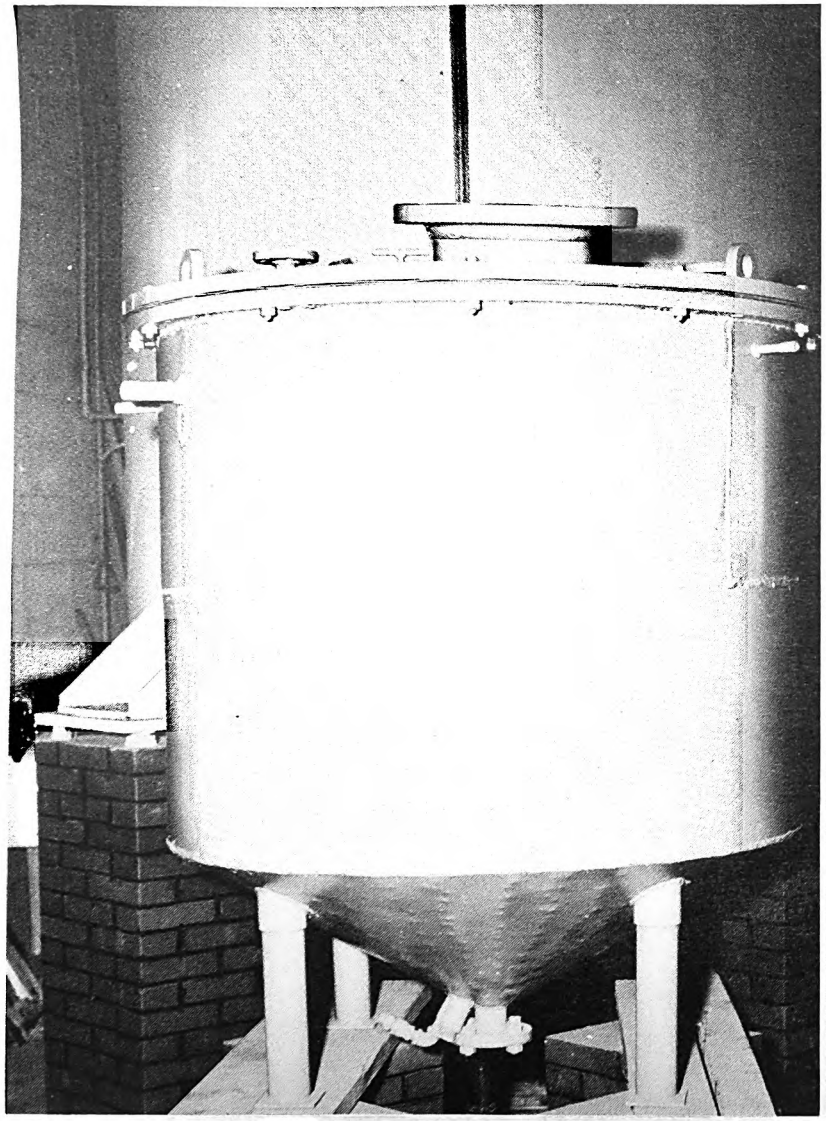

Figure 3. - 250-gallon, zirconium-lined, jacketed reactor.

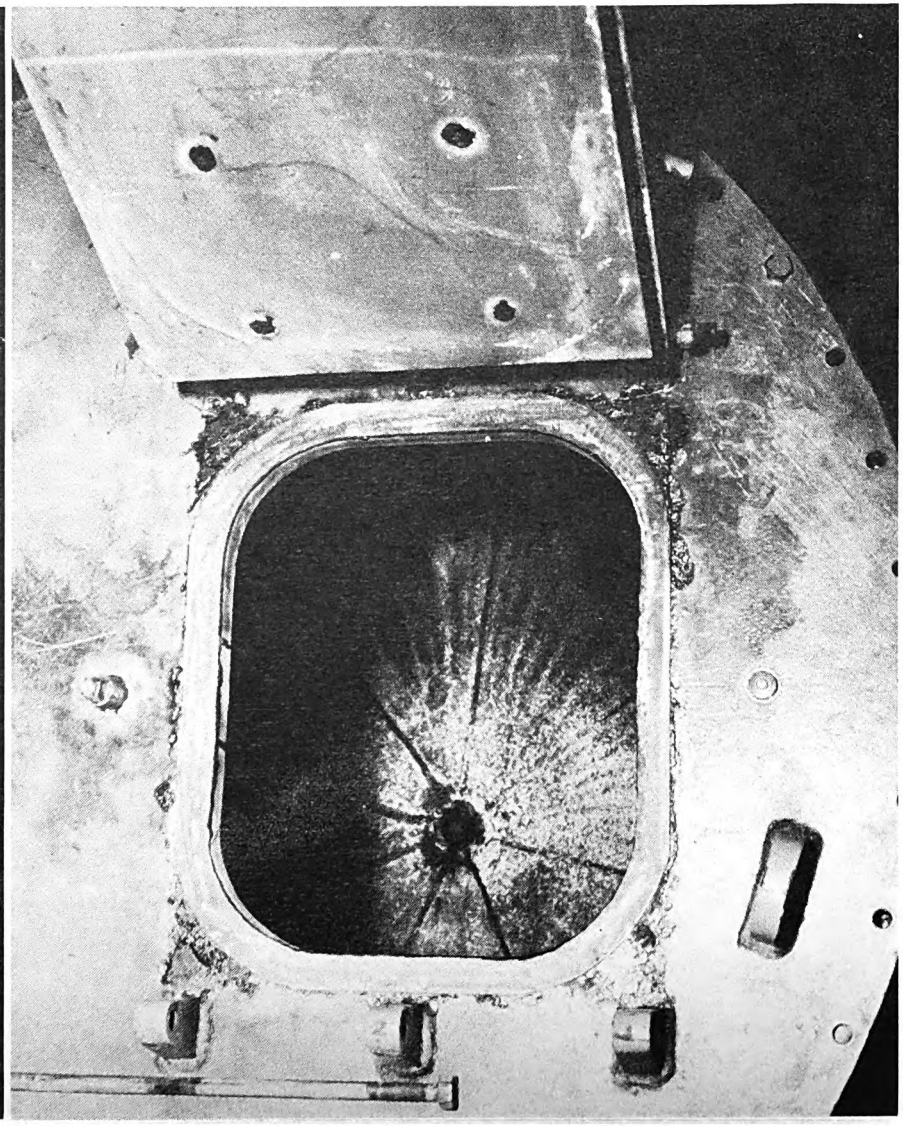

Figure 4. - View looking into zirconium-lined tank. Note zirconium-faced manhole and pie-shaped segments on bottom of tank.

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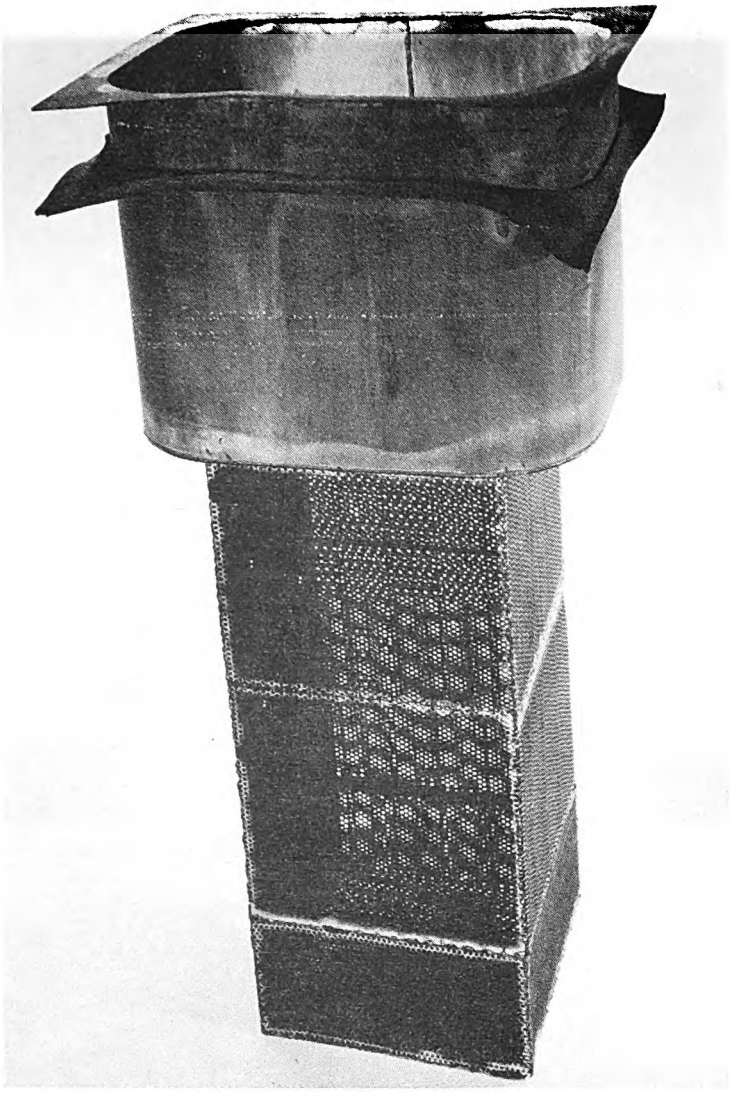

Figure 5. - Basket strainer fabricated of sheet and perforated zirconium sheet for use in zirconium-lined tank.

Figure 6. - Zirconium agitator after 3 months' service in zirconium phthalate solutions. Shaft is made of extruded zirconium sponge, and wheel is fabricated from sheet and ingot stock.

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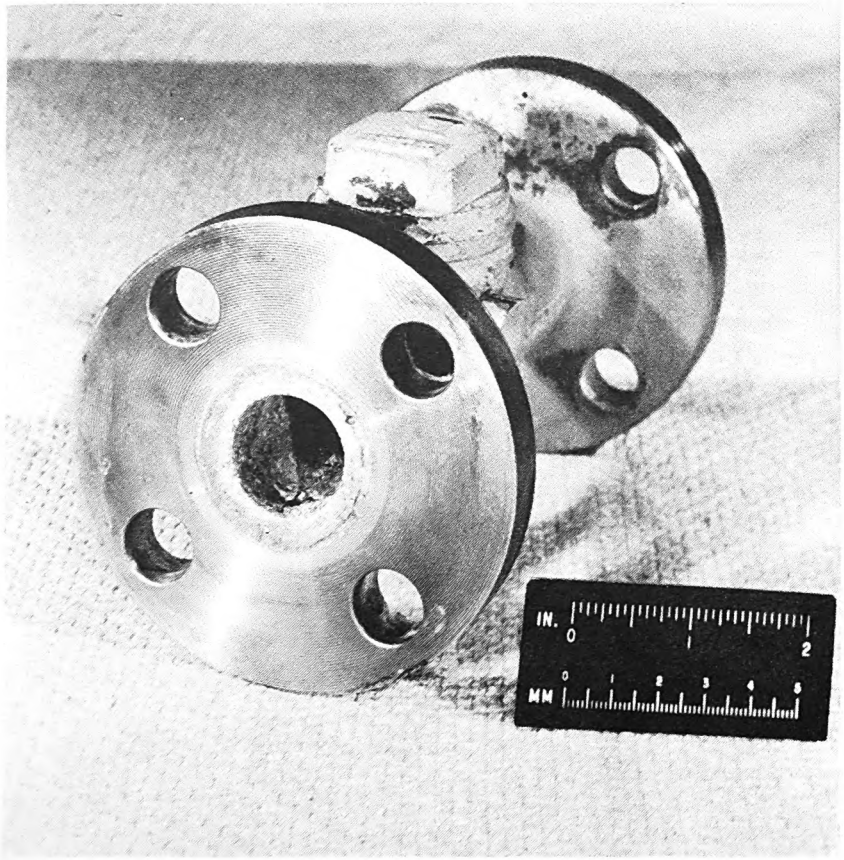

Figure 7. - Type 316 stainless-steel horizontal check valve, which failed in zirconium phthalate solutions after 3 weeks.

Figure 8. - Glass-covered steel agitator, which failed in service handling zirconium phthalate solutions.

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Corrosion Test Bomb

This bomb (fig. 2) was patterned after standard steel bombs for use in special corrosion studies involving the use of caustic and acids, by K. B. Higbie.[10] The design pressure is 2,200 to 2,400 p.s.i. at temperatures not to exceed 650° F.

PRODUCTION APPLICATIONS

General

The production of zirconium may be broken down into several well-defined unit processes. In each step the corrosive materials handled are of a different nature and require a specific description of the solutions, agents, and conditions encountered.

To present a clear explanation of the applications shown, it is necessary to make use of captions and isolated paragraphs. Reference is made to similar situations to avoid repetition.

Zirconium-Lined Tank

This tank (figs. 3 and 4) was designed by K. E. Royer[11] to handle residues resulting from the purification and reduction of zirconium. It was believed that a glass-lined tank would be unsuitable for this purpose because of the abrasive qualities of the slurries to be handled.

In this operation, residues containing zirconium tetrachloride, zirconium oxide, and tramp materials are fed into water through a zirconium basket (fig. 5). Hydrochloric acid is formed during the reaction, so that the resulting slurry is strongly acidic. Heat is given off, resulting in slurry temperatures up to 150° F.

This vessel was used temporarily to replace a glass-lined tank, which failed in the high-speed agitator. After 3 months in this solution, no evidence of corrosion was noted.

Basket Strainer

This strainer fits the manhole of the tank described above. It is used to keep tramp material out of the tank and minimize plugging of lines between the tank and filter press.

High-Speed Agitator

Figure 6 shows a zirconium agitator after 3 months' service in zirconium phthalate solutions. The shaft is made of extruded zirconium sponge, and the wheel is fabricated of arc-melted sheet and ingot stock. No corrosion was noted at the time this was taken out of service.

In this reaction zirconium phthalate is precipitated from zirconyl chloride solution by the addition of diammonium phthalate. The pH of this reaction is adjusted to 1.5 - 2.0 with hydrochloric acid and maintained at 165° - 190° F. The corrosive nature of this solution is due to the presence of hydrochloric and phthalic acids and is further enhanced by trace quantities of ferric and other chlorides.

10/ Chemical engineer, Northwest Electrodevelopment Laboratory, Bureau of Mines, Albany, Oreg.

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Evidence of the corrosive properties of this solution is offered in figure 7, which shows a type 316 stainless-steel valve that failed in service after 3 weeks. Figure 8 shows a glass-covered steel agitator that also failed in this service.

pH Electrode Assembly

The assembly shown in figure 9 was fabricated from ingot zirconium to replace a standard type 316 stainless-steel unit (fig. 10), which failed after 10 months' service. This device is suspended in a weir box leading to a rubber-covered rotary vacuum filter. In normal operation, about half of the electrodes are submerged in the zirconium phthalate solution described in the previous application.

Steam-Jet Exhauster

This exhauster was fabricated to replace a 10-stage blower, which could not be kept in serviceable condition, requiring overhaul at 10-day intervals. The impellers of this blower were type 316 stainless steel, and the housing was cast iron. Fumes entering the blower had been passed through a scrubbing tower and a second trap containing iron turnings to remove the hydrochloric acid.

In making up zirconium chloride feed solutions, the chloride is added to the tank through a dip pipe by suction. Strong hydrochloric acid and zirconium chloride fumes evolved during this addition must be handled by the evacuation system.

The steam jet pictured in figure 11 was put into use on February 1, 1953 and was still giving trouble-free performance 1 year later.

The throat piece from a cast-iron steam ejector is shown in figure 12. This is shown as evidence of the corrosive quality of the exhausted gases.

Zirconium Thermowell

Two thermowells were fabricated of tubing and sheet zirconium for use in the zirconium chloride feed-solution makeup tanks. These replaced glass-covered thermowells. The tube shown in figure 13 now has had 20 months in this service. A second unit has about the same length of service, 7 months in zirconium phthalate solutions.

The plant-feed solutions are made up to contain 1 pound of zirconium per gallon and are 1 normal in hydrochloric acid. Temperatures are maintained at less than 150° F.

Zirconium Saddles

The saddles, clamps, and machine bolts shown in figures 14 and 15 were fabricated from Zirconium to effect temporary repairs to glass-coated Pfaudler agitators in use in feed-makeup tanks (described in the preceding section).

The failures were due to the sandblasting action on the glass lining by solids in the solutions and subsequent acid attack. Repairs were made by plastering furane resin over the exposed metal, wrapping with teflon strip, and clamping into place with the saddles described. These repairs were temporary, lasting about 1 month.

Agitator Hub

After repeated failure of the temporary repair noted in the preceding section, the hollow impeller shaft was cut off about 3 feet above the anchor-type arms and a

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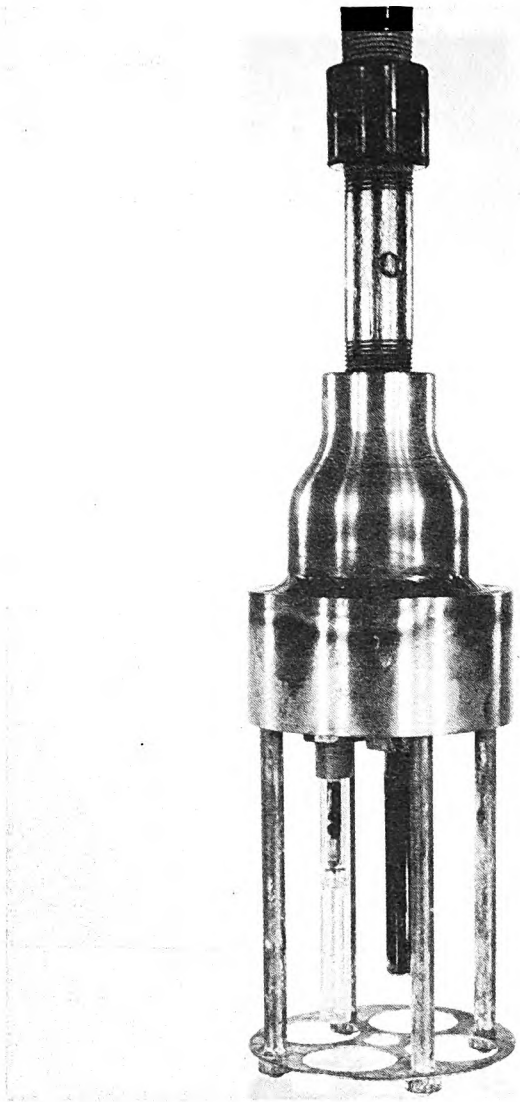

Figure 9. - Zirconium pH electrode assembly fabricated to replace one shown in figure 10. The support rods were in use 10 months.

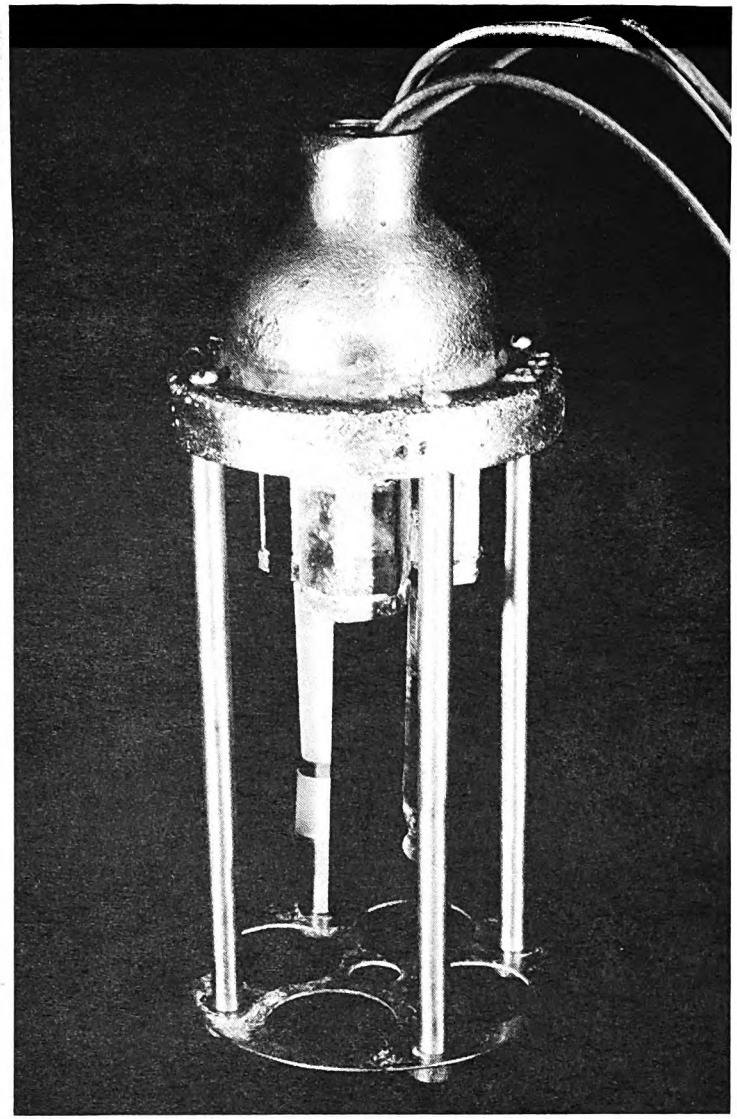

Figure 10. - Type 316 stainless-steel pH electrode assembly after 10 months' service in zirconium phthalate slurry. The rods supporting the bottom guard are new.

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Figure 11. - Exploded view of steam jet, showing integral parts of ejector, all of which are made of zirconium, except hand-wheel.

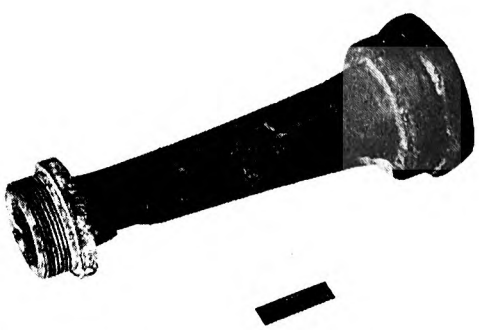

Figure 12. - Cast-iron throat piece from steam ejector after 1 week service.

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Figure 13. - Zirconium thermowell used in feed-makeup tank.

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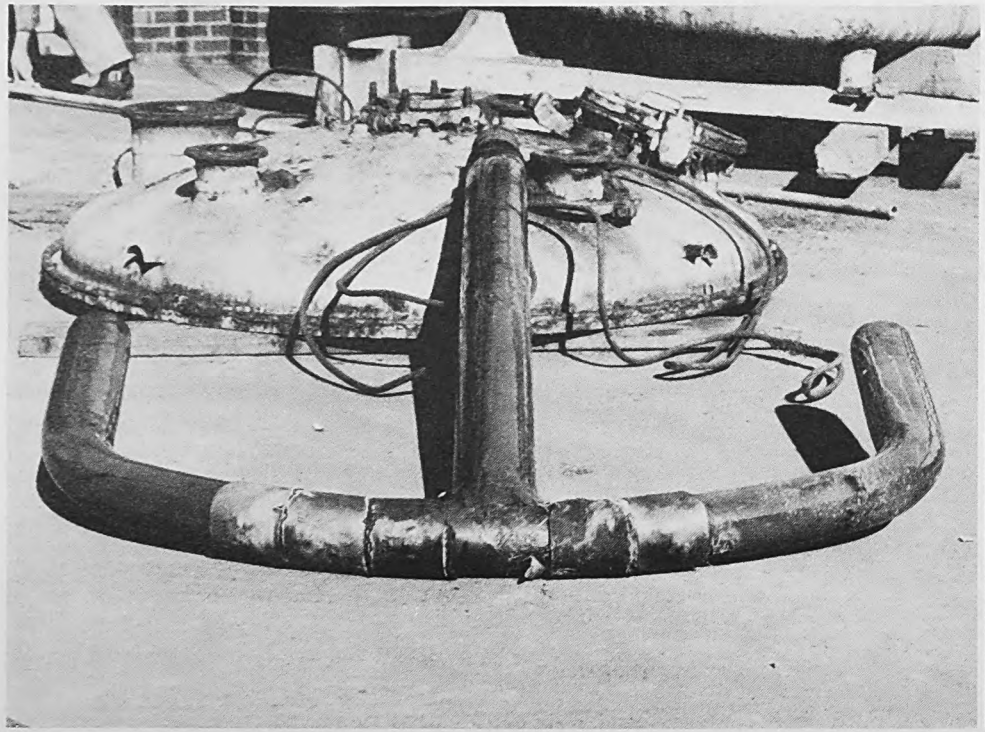

Figure 14. - Glass-coated agitator from 500-gallon reactor, showing 5 zirconium saddles.

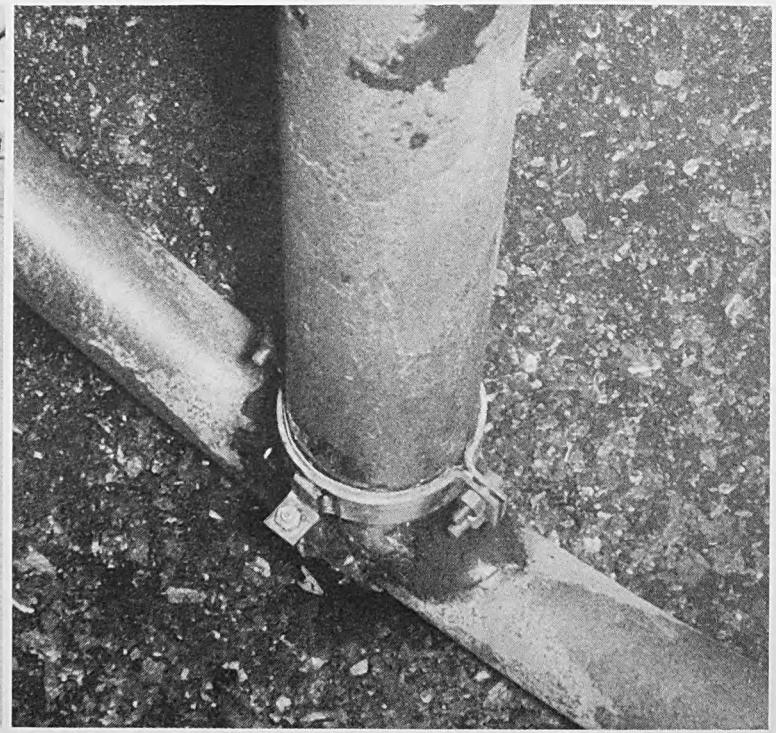

Figure 15. - View of interior of glass-lined Pfaudler kettle, showing zirconium clamp and saddle used in making temporary repair to agitator. Vertical shaft is about 5 inches in diameter.

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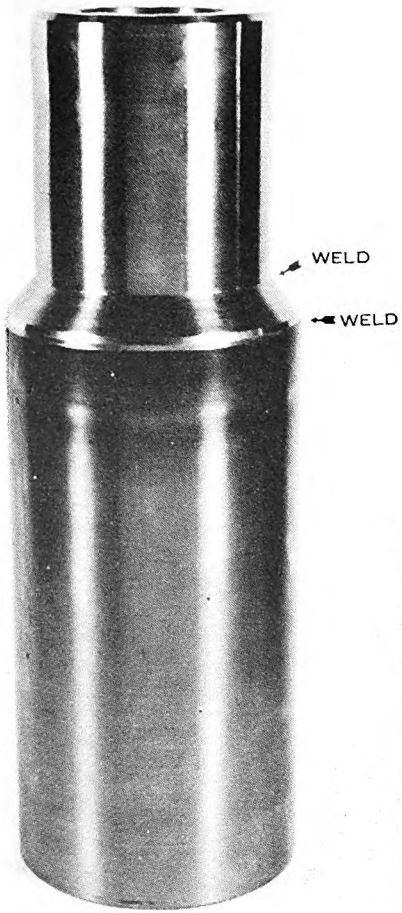

Figure 16. - Zirconium hub for wood agitator that replaced one shown in figure 14. Hub is about 5 inches in diameter.

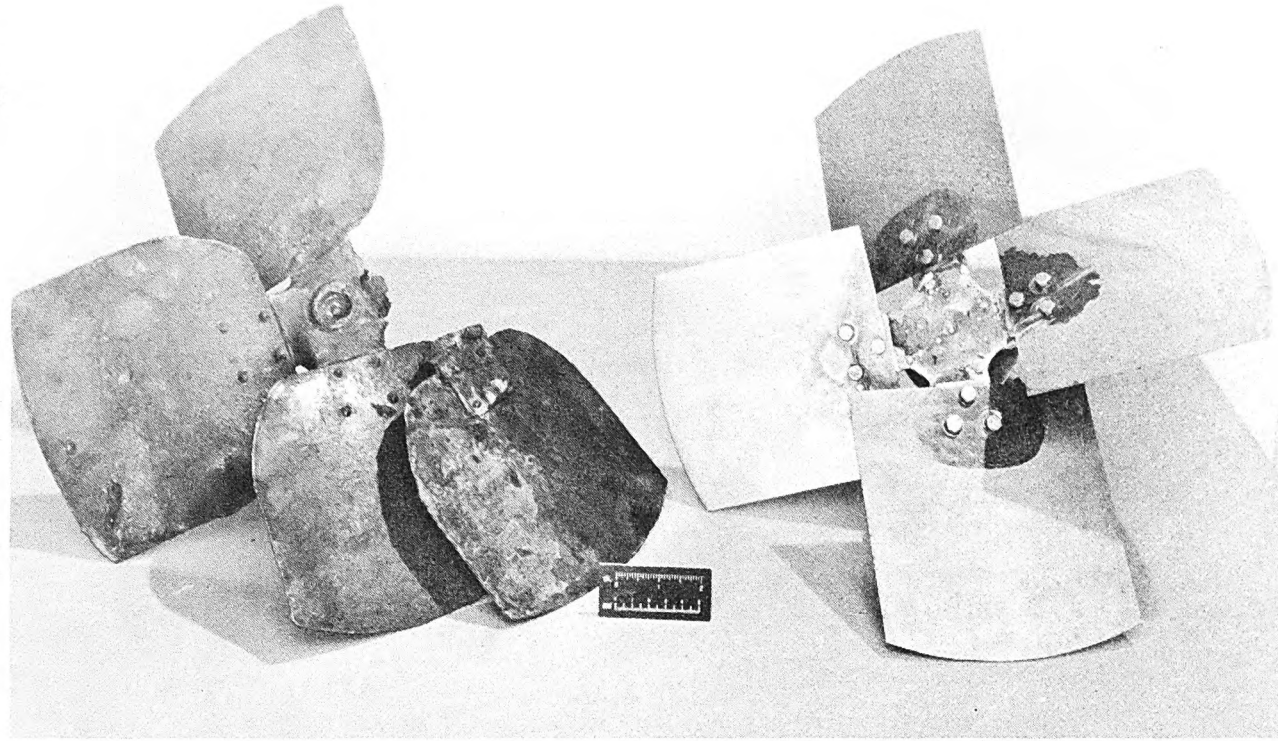

Figure 17. - On left—plastic-coated steel exhaust fan that failed after 8 months exhausting hydrochloric acid fumes from feed filter press. On right—zirconium replacement fan; this fan now has had 10 months' service without visible deterioration.

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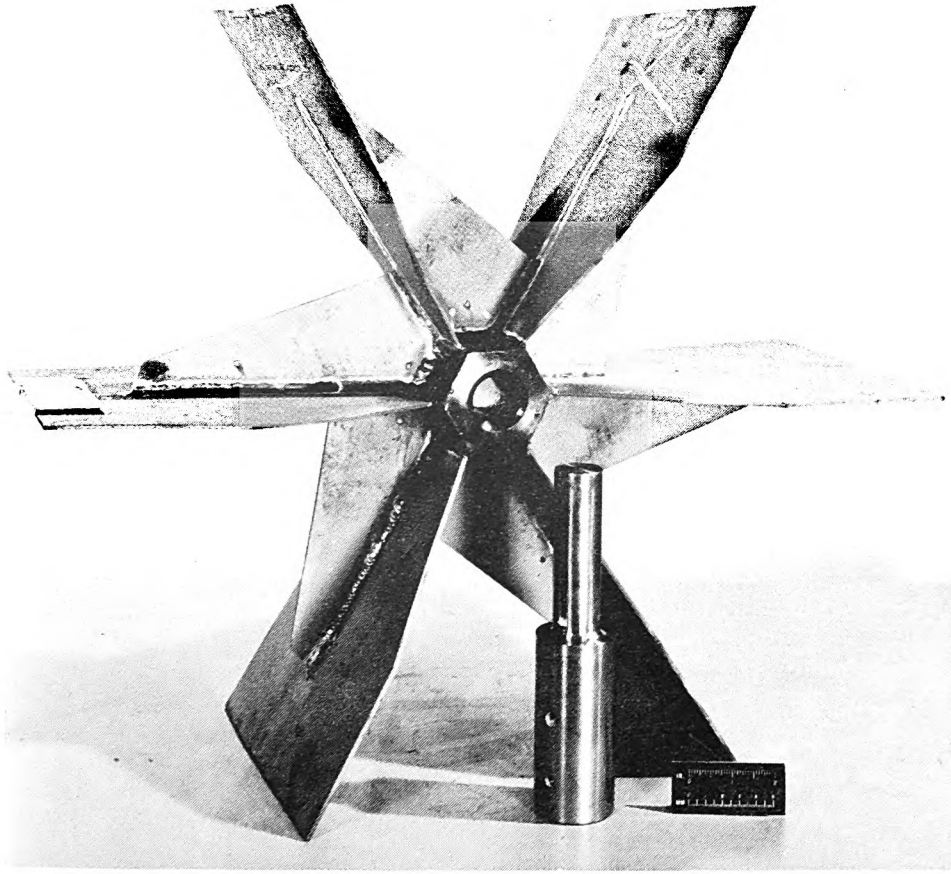

Figure 18. - Zirconium fan wheel fabricated to replace one shown in figure 19. This wheel now has had 8 months' service.

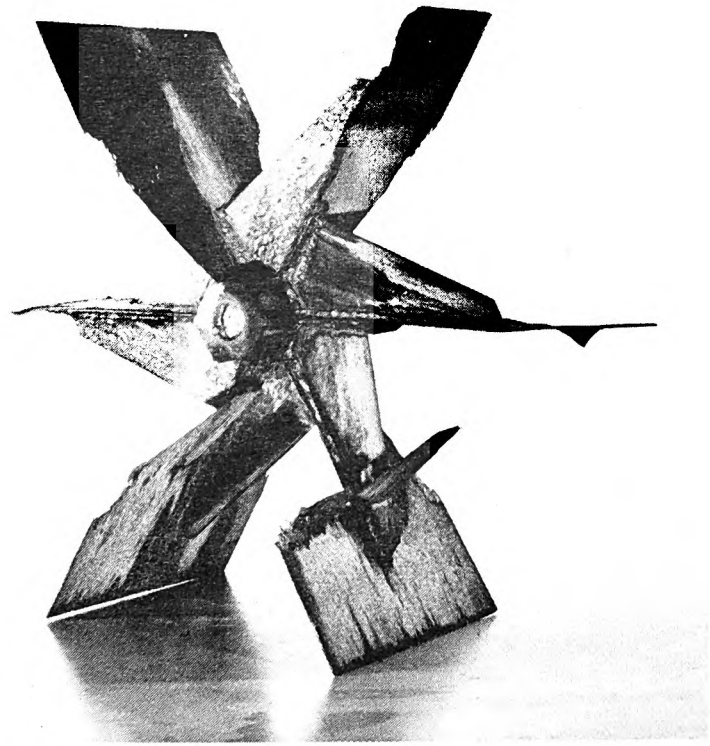

Figure 19. - Mild-steel fan wheel from blower exhausting fumes from the chlorination area, after 2 months' service. Fumes handled are mainly wet hydrochloric acid.

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Figure 20. - Zirconium mechanical seal shown on shaft of a rotating element.

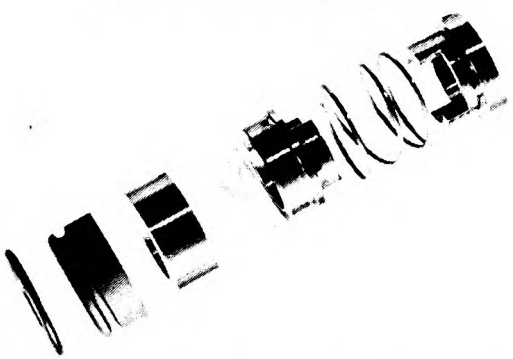

Figure 21. - Exploded view of zirconium mechanical seal. From left to right, rubber "O" ring, graphite rub ring, zirconium rub plate, teflon seal, zirconium pressure plate, zirconium spring, and zirconium driving plate.

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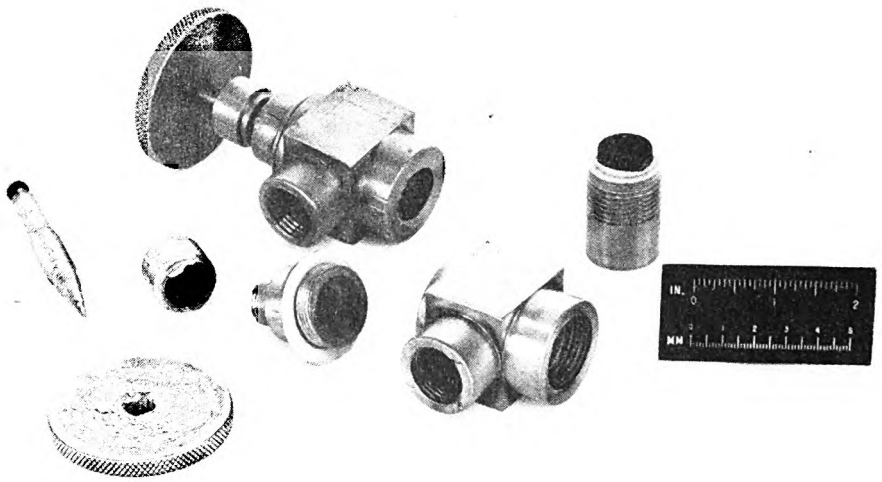

Figure 22. - Zirconium needle valve used to regulate flow of concentrated hydrochloric acid to rotary filter.

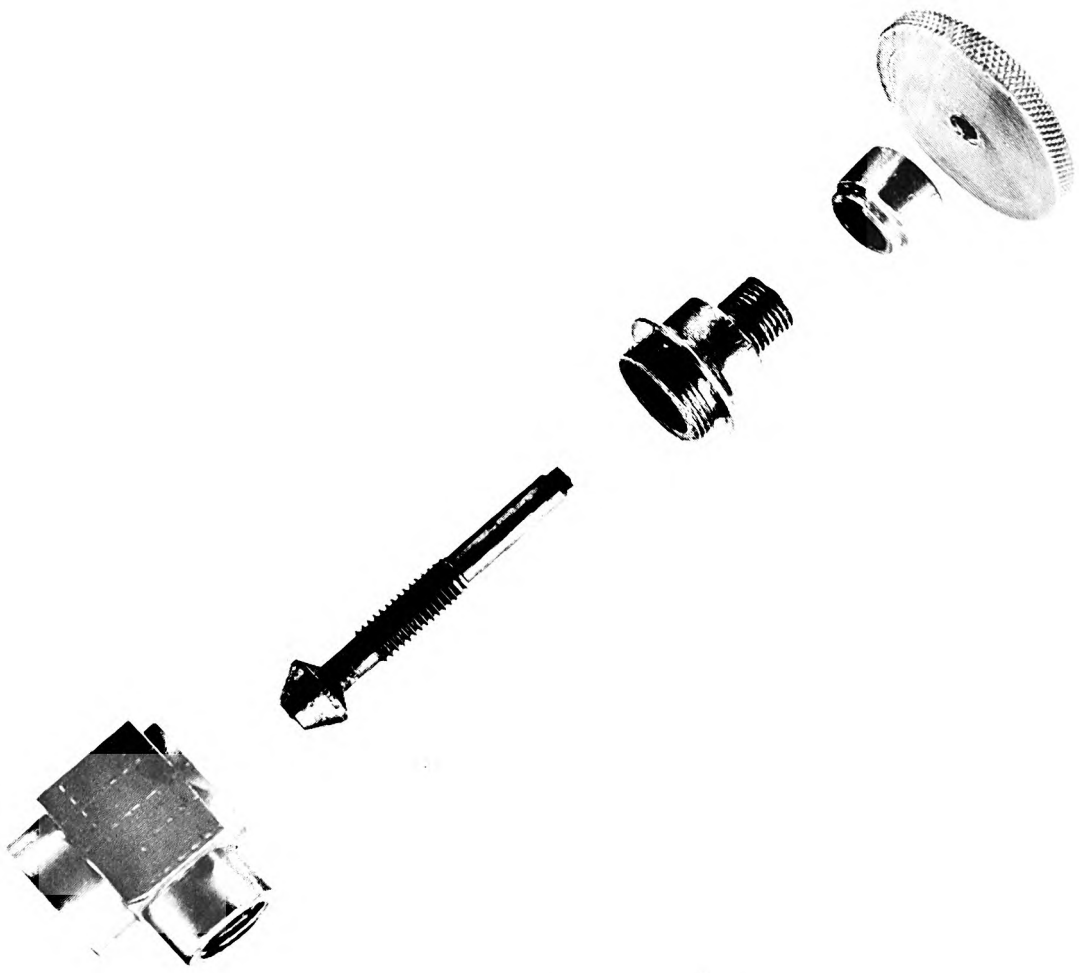

Figure 23. - Exploded view of globe valve.

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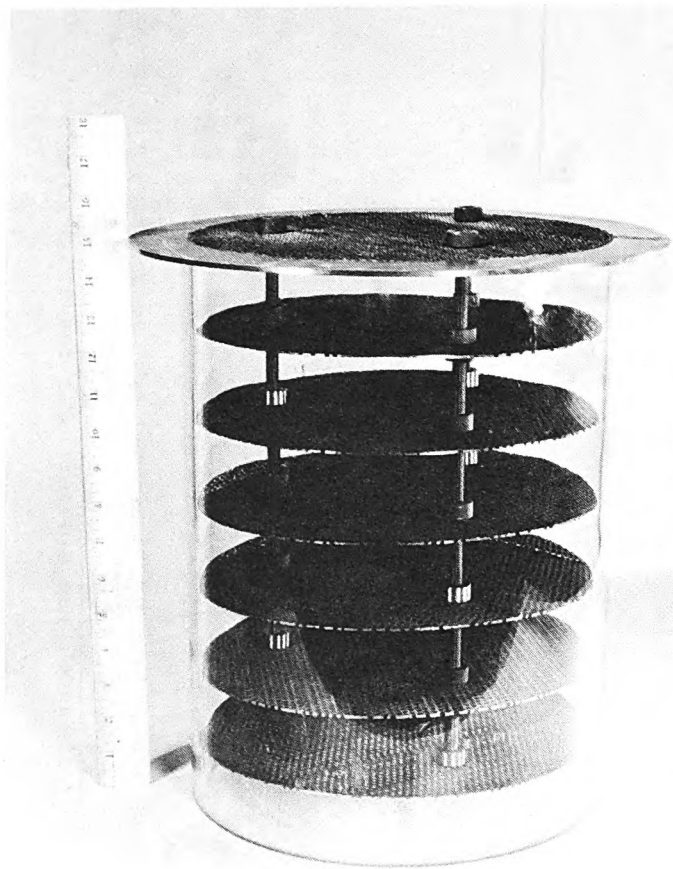

Figure 24. - One section of 10-inch-diameter pulse column now being installed as an extractor. Plates, bolts, and spacer nuts are zirconium.

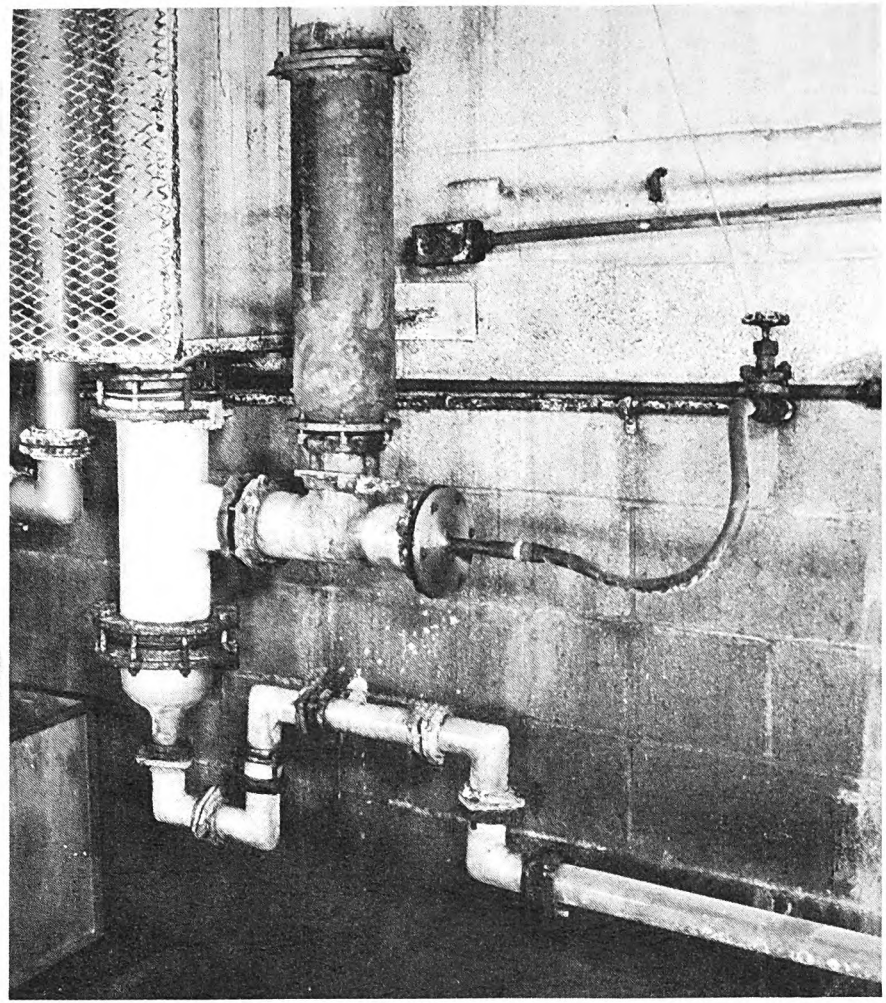

Figure 25. - Zirconium flange near base of noxious-gas scrubber in chlorination plant is in contact with wet chlorine and hydrochloric acid fumes.

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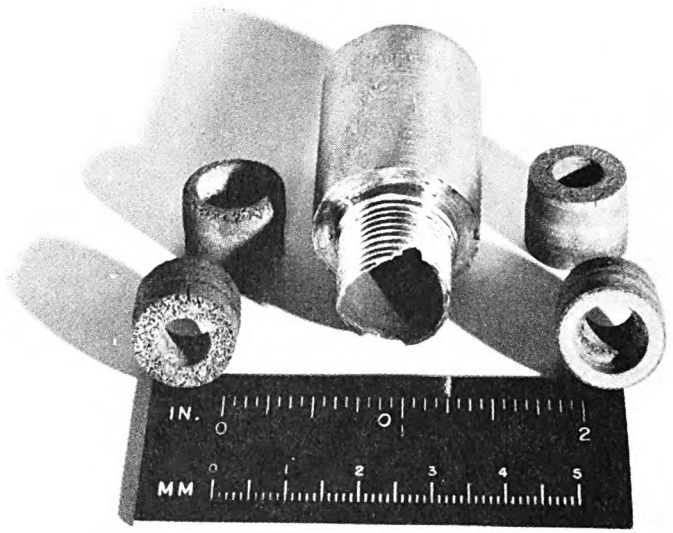

Figure 26. - Hastelloy ball seats from Milton Roy pump handling concentrated hydrochloric acid. Length of service, about 3 months. Zirconium seats were found to be too ductile for this service. Zirconium nipple shown failed in same service. Source of zirconium is not known.

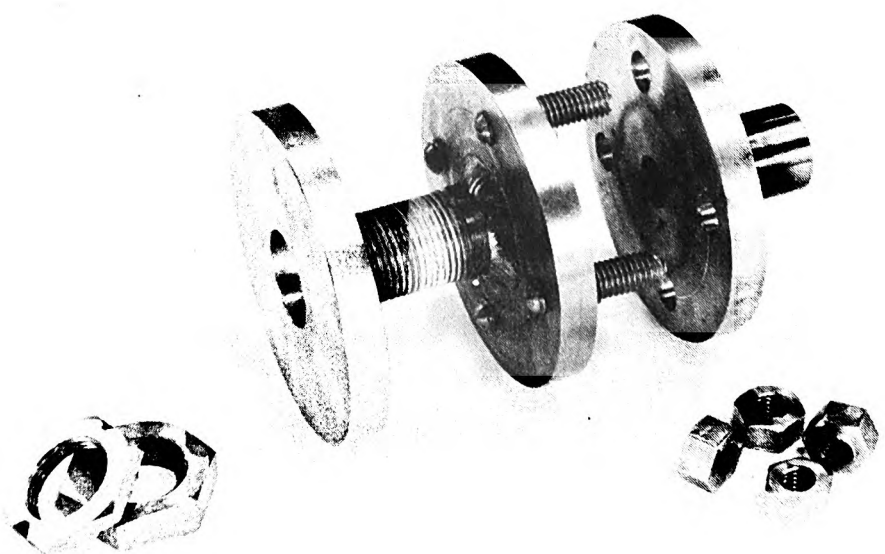

Figure 27. - Zirconium flanges and adapter parts for nozzle on rubber-lined acid tank.

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wooden impeller fitted to it. This impeller, which was bolted to the shaft with zirconium bolts, is still in service; however, the remainder or hub of the original agitator failed through corrosion.

The zirconium hub shown in figure 16 was fabricated from two ingots and a disk. This hub now has had about 11 months service.

Exhaust Fan

A zirconium fan was fabricated to replace a plastic-coated fan that failed after 8 months' use. This fan exhausts hydrochloric acid fumes from a feed filter press. The zirconium unit shown in figure 17 now has had about 10 months' service without visible signs of corrosion.

A second zirconium exhaust fan (fig. 18) now has had 8 months' service exhausting fumes from the direct chlorination area. Corrosives are handled as wet hydrogen chloride. A mild-steel blower after 2 months in this service is shown in figure 19.

Mechanical Seal

A mechanical seal designed by W. A. Aschoff[12/] was fabricated for use on centrifugal pumps. Several good seals are available, but none of them are easily adapted to pumps made of high-silicon cast iron, since they require that the inside surface of the packing glands of the pumps be machined by grinding to attain a suitable fit or surface.

The first seal, shown in figures 20 and 21, was put into use on February 17, 1953, and now has had 12 months of satisfactory service. About 20 of these are now in use.

Needle Valve

A zirconium needle valve was fabricated to regulate the flow of concentrated hydrochloric acid to a rotary filter. This valve, shown in figure 22, was in use about 3 months. Some corrosion was noted on the valve seat, but the valve was still serviceable.

Globe Valve

A zirconium globe valve (fig. 23) is used to regulate the flow of gas being evacuated from furnaces in the reduction unit. The fumes are essentially hydrochloric acid. Length of service is estimated at 2 years.

Pulse-Column Plates

Zirconium bolts, nuts, and perforated pulse plates are used in a 10-inch-diameter pulse column now being erected. This column, designed by R. H. Nielsen,[12/] will be approximately 35 feet high and will process feed solutions being contacted with acidified solvent. One section of this column is shown in figure 24.

Zirconium Pipe Fittings

Zirconium pipe fittings and flanges are being used in many services through the process without failure. A notable exception is pictured in figure 26, which

12/ Chemical engineer, Northwest Electrodevelopment Laboratory, Bureau of Mines, Albany, Oreg.

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shows a zirconium nipple that failed after 3 months' service in concentrated hydrochloric acid. The source of the metal used is not known, but it is probably made from graphite-melted ingot. Other typical examples are shown in figures 25, 27, and 28.

Spray Nozzles

The use of zirconium spray nozzles in noxious gas scrubbers is an important application, as they can be fabricated easily from bar stock and are resistant to most corrosive gases encountered.

The first nozzles installed in 1949 are still serviceable after use in wet chlorine and hydrochloric acid fumes. The comparative resistance of nickel and zirconium is shown in figure 29.

Interface Control Mechanism

In June 1952, N. F. Hyde, a laborer in the purification plant, proposed an interface control device that did not depend upon rubber or other flexible tubing. The use of zirconium in this application, as indicated in figure 30, made possible the immediate adaptation of this suggestion. The zirconium rods have had approximately 14 months' service without evidence of corrosion. Corrosives handled are 3.5 normal hydrochloric acid and 5 normal sulfuric acid.

Water Aspirator

In 1950 when the large-scale zirconium reduction unit was placed in operation, there was one brass aspirator on each purification furnace. These were gradually replaced with zirconium units, so that 12 are now in operation, with up to 3 years' service.

These aspirators, used to evacuate purification and "PR" furnaces, are in contact with hydrochloric acid fumes. An exploded view of an aspirator is shown in figure 31.

Surge Bottle

The surge bottle serves as a trap in the evacuation system described above. The aspirator suction line is fastened to the bottom hose fitting, with the furnace evacuation line fastened at the top of the bottle. On occasion, low water pressure at the aspirator permits a higher pressure at that point, so that water flows from the aspirator into the furnace.

The new surge bottle shown in figure 32 permits constant observation of the flow of gas in the system.

Hydraulic Valve Part

A zirconium hydraulic valve (fig. 33) was put into use in hydraulic systems on forklift trucks. Standard mild-steel valves furnished on the original equipment were found inoperative after 2 weeks' use, while the zirconium valve spools are still in service after 2 years.

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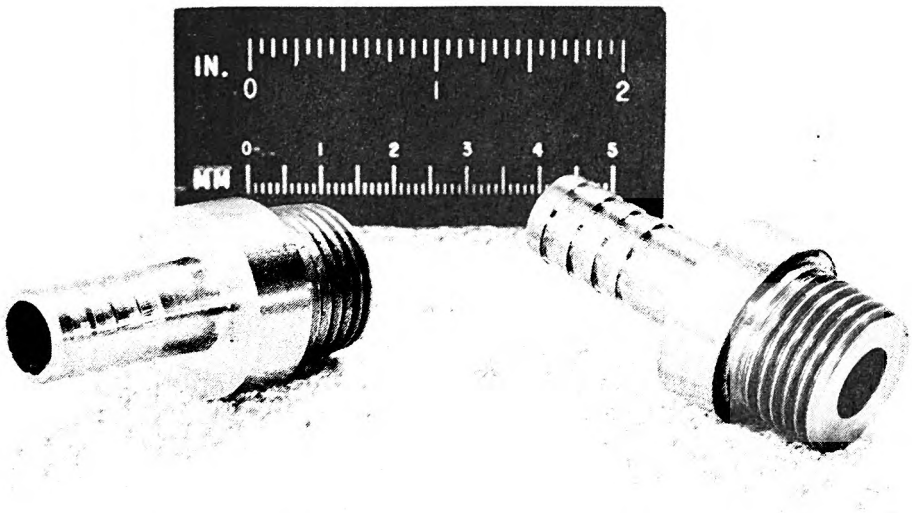

Figure 28. - Male adapters for use with polyethylene tubing in concentrated hydrochloric acid service.

Figure 29. - Zirconium spray nozzles, couplings, and pipe after 14 months service in exit-chlorine scrubbing tower, in contact with wet chlorine and chlorides of hydrogen, iron, aluminum, silicon and titanium. Top pipe is pure nickel after 6 weeks' service.

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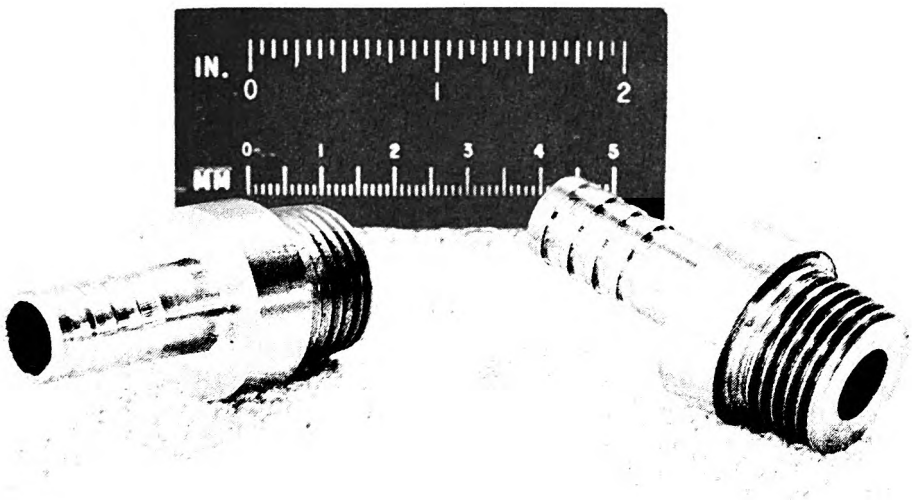

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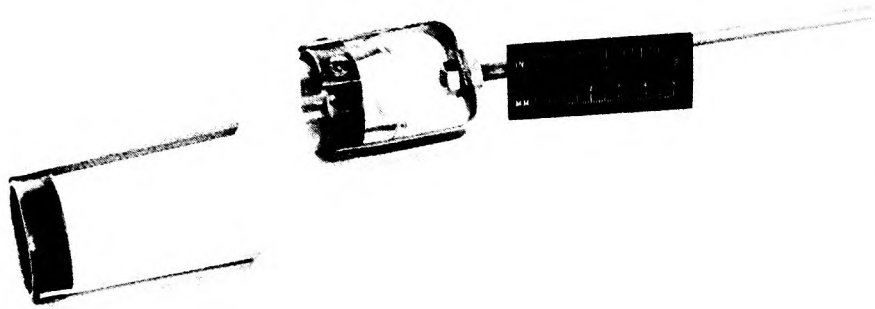

Figure 30. - Zirconium clamp on 1-inch glass pipe and rod used to raise and lower column-interface control mechanisms.

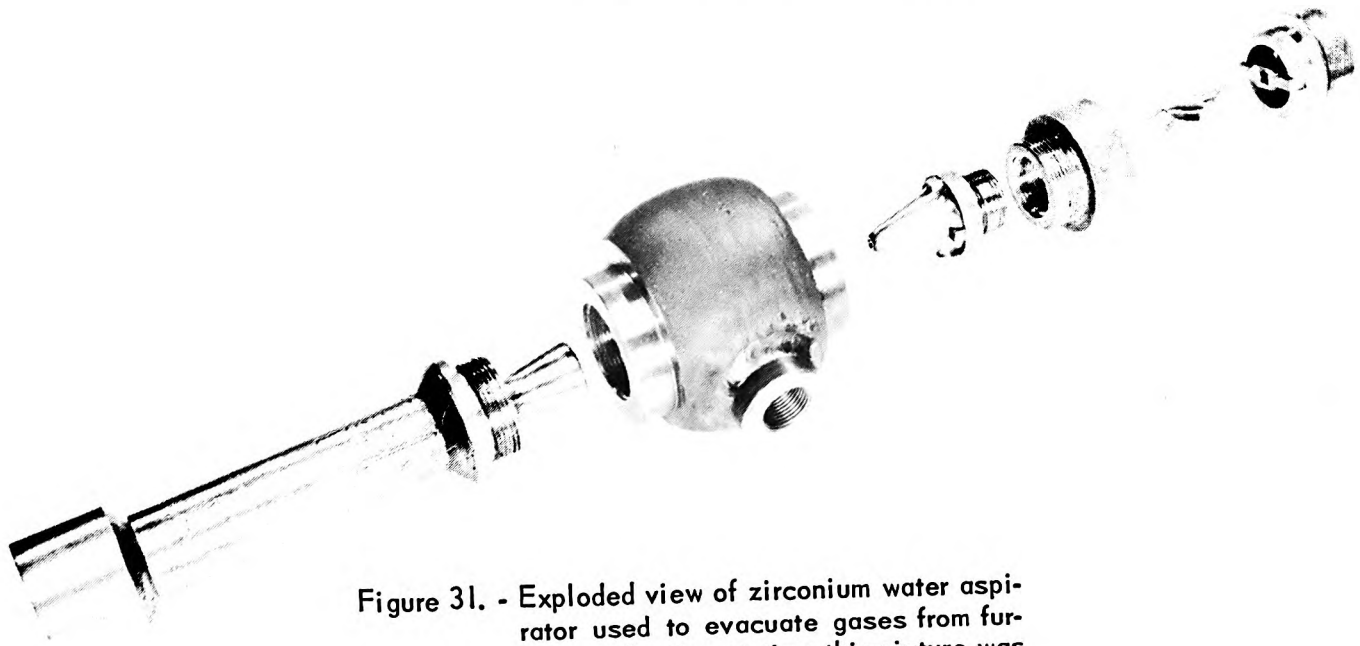

Figure 31. - Exploded view of zirconium water aspirator used to evacuate gases from furnaces. Service at time this picture was taken exceeded 3 years.

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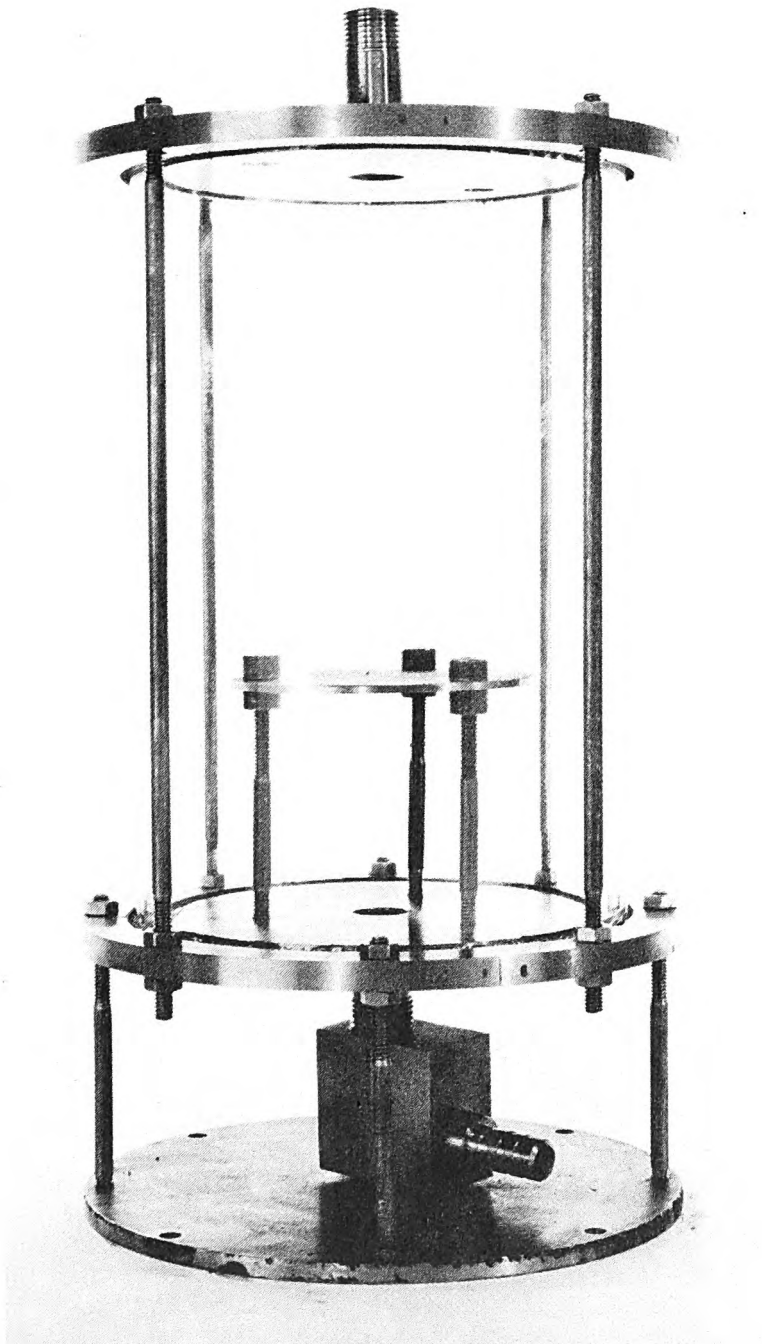

Figure 32. - Zirconium and glass surge bottle for use in the evacuating systems on purifications and "PR" furnaces.

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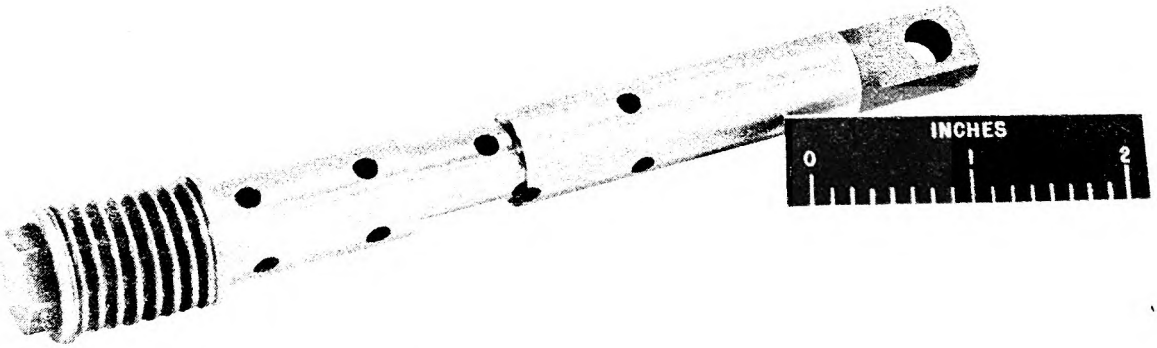

Figure 33. - Zirconium hydraulic valve for use on fork lift trucks.

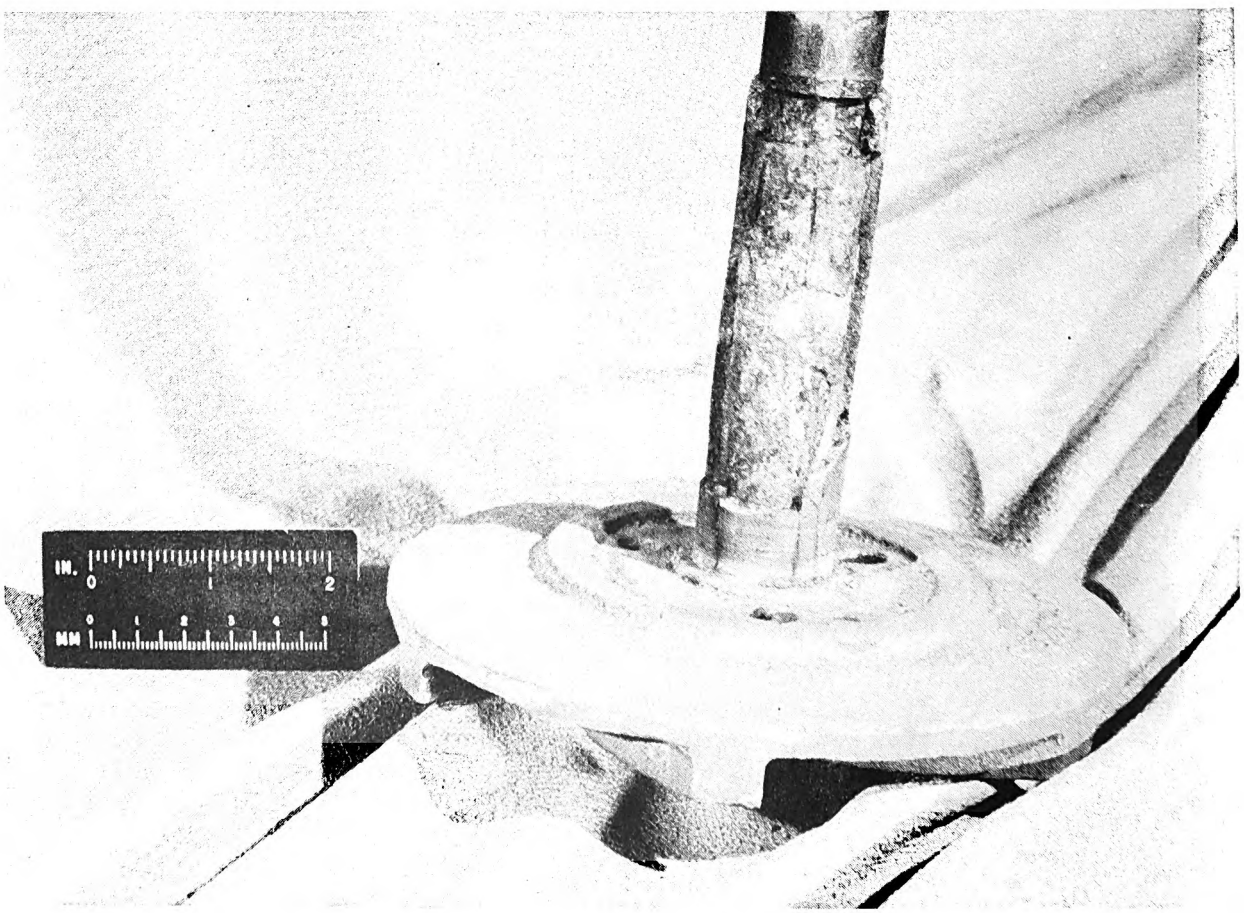

Figure 34. - Rotating element, showing appearance of shaft under shaft sleeve.

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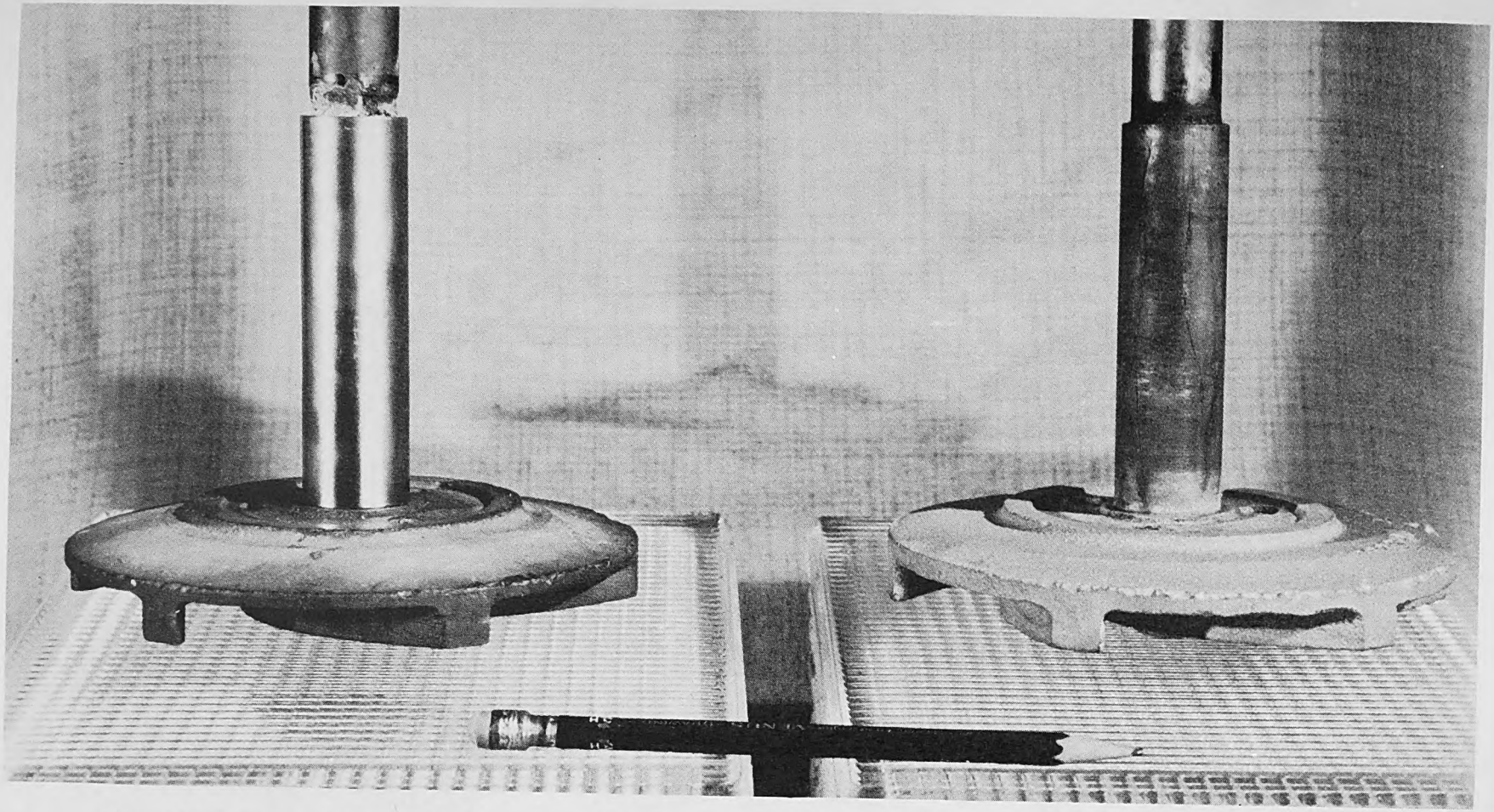

Figure 35. - Two rotating elements from centrifugal pumps. On left–zirconium shaft sleeve as pressed to steel shaft and sealed with plastic. On right–standard shaft sleeve, showing cracks resulting from corrosion.

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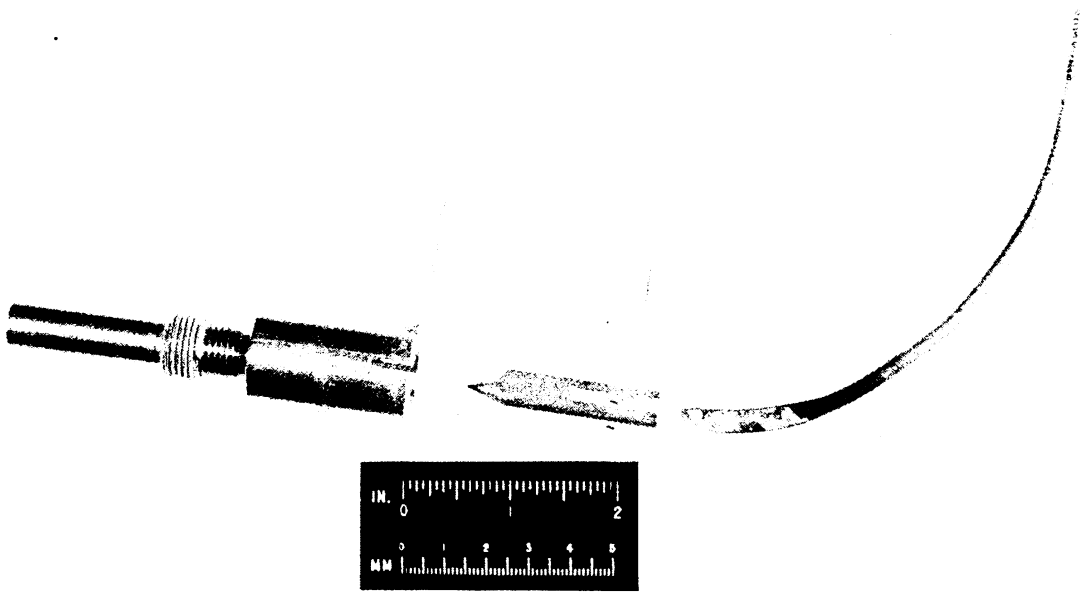

Figure 36. - Exploded view of constant-level valve.

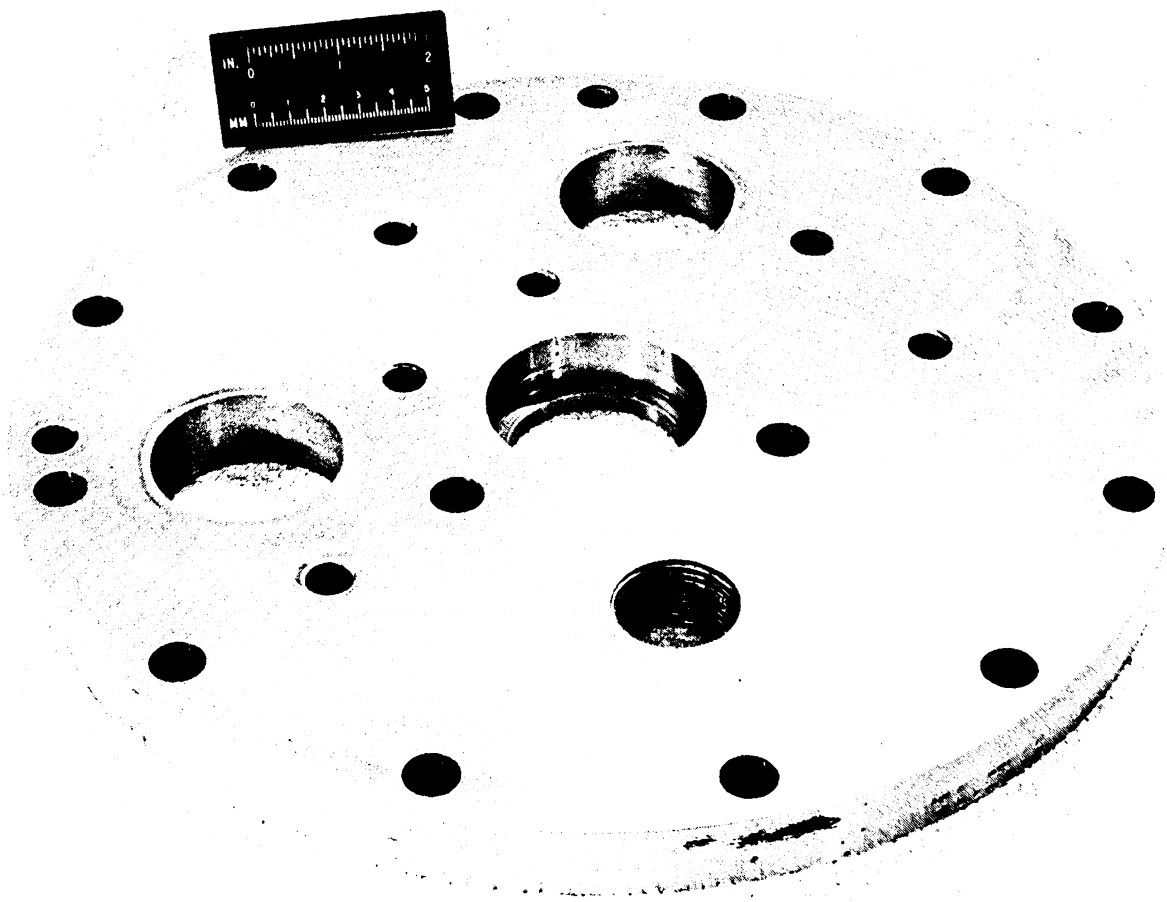

Figure 37. - Rotary-filter valve plate.

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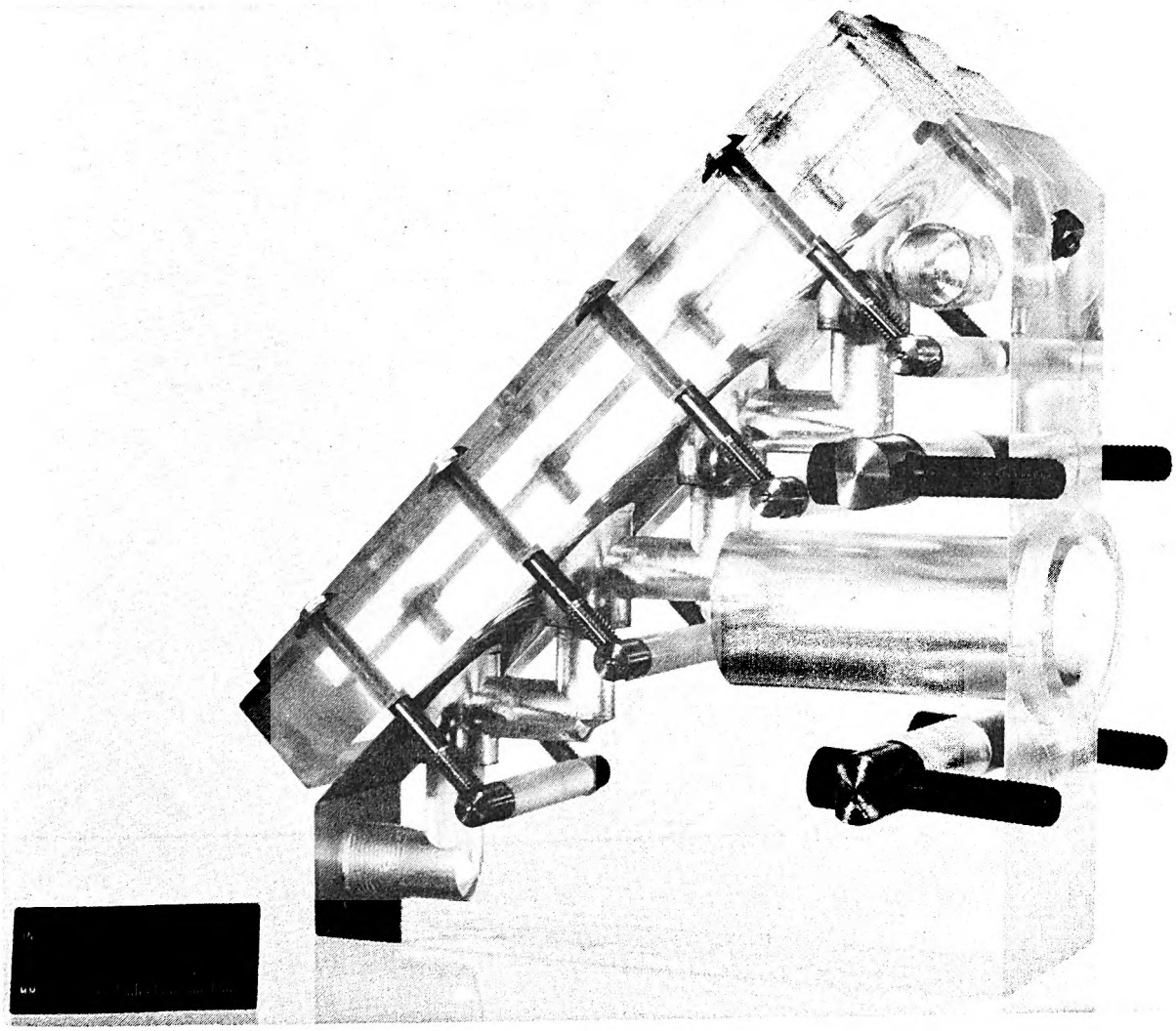

Figure 38. - Liquid end of plastic metering pump, showing use of zirconium bolts, nuts, and studs.

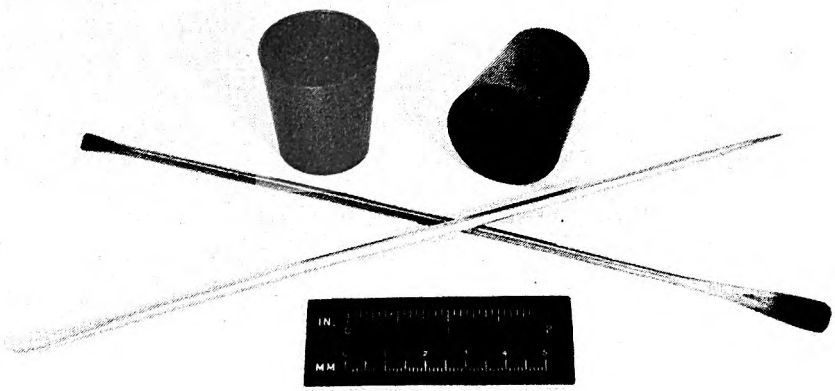

Figure 39. - Zirconium crucibles after eight caustic fusions; zirconium spatulas.

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Shaft Sleeve

High-silicon cast-iron shaft sleeves on the rotating element of centrifugal pumps failed owing to corrosion of the steel shaft under the shaft sleeve. The mortality rate is quite high; 9 of 15, or 60 percent, of the pumps in 1 service have failed in 17 months of service.

Replacement of these shaft sleeves is done ordinarily at the factory, at approximately the cost of totally new elements. A badly corroded shaft is shown in figure 34.

The zirconium shaft sleeves pictured in figure 35 have not had any length of service at this time but are expected to have a comparatively longer life, because of their higher tensile strength if corrosion takes place on the steel shaft.

Zirconium shaft sleeves may not be suitable where packing is used, since the ductile metal does not resist abrasive wear.

Float Check Valve

This valve was fabricated to maintain a constant level in a solution head tank. It was in service 18 months, handling zinc sulfate solutions 1/3 molar in sulfuric acid. The plastic float used with this valve is not shown in figure 36, which illustrates the valve parts.

Rotary Filter Valve

A zirconium face plate, shown in figure 37, supports a hard-rubber valve on a 3 by 6 rotary filter, which is handling hot zirconium phthalate solutions (described under "High-Speed Agitator"). The temperature of about 160° F. tends to warp the valve, so that leakage occurs between the rub plate and the rotary valve. This warpage is avoided by use of the zirconium backing or face plate.

Proportioning Pump Parts

Zirconium has been used successfully in repairing positive-displacement proportioning pumps. Ceramic plungers have been replaced with zirconium in several locations where hydrochloric acid, varying from 1 to 4 normal, is being pumped.

Several Plexiglas Milton Roy-type pumps have been equipped with zirconium plungers and the liquid ends bolted together with zirconium bolts, nuts, and studs. Figure 38, an assembled liquid end, shows the use of studs, bolts, nuts, and pins in this application. Four black-iron machine screws are shown being used in an easily accessible location on the liquid end.

LABORATORY APPLICATIONS

Zirconium crucibles are being used at the Northwest Electrodevelopment Laboratory for caustic fusions, where zirconium contamination is not objectionable and an iron determination is being made. Iron crucibles have an expectancy of 1 to 5 fusions. The zirconium crucibles are expected to last for a minimum of 25 fusions.

The utility of zirconium in analytical laboratories is outstanding. Probably the most important use is for caustic fusions where platinum and other wares corrode and fail. The crucibles pictured in figure 39 show only slight corrosion after eight caustic fusions.

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